REPORT

on

MASTER

EXPERIMENTAL SCREENING OF CARBON-BASE MATERIALS
FOR IMPACT MEMBERS IN ISOTOPIC HEAT SOURCES

by

G. K. Bansal and W. H. Duckworth

November, 1976

Prepared for the Division of Nuclear Research and Applications,
U.S. Energy Research and Development Administration,
Under Contract W-7405-eng-92, Task 67

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FOREWORD

The research described in this report was conducted as a part of a technology program concerned with selection of improved impact member and heat shield (ablative) materials for isotopic heat sources. The heat sources are used in static and dynamic power conversion systems. It was sponsored at Battelle's Columbus Laboratories by the Energy Research and Development Administration's Nuclear Research and Applications Division (NRA) under Contract W-7405-eng-92, Task 67. A. P. Litman and W. M. Pardue are serving as Program Managers, respectively, at ERDA/NRA and Battelle-Columbus. F. L. Bagby served as the Battelle-Columbus Program Manager until March, 1976. In addition to the authors and Program Managers, W. C. Chard, F. A. Simonen*, D. E. Niesz and J. F. Schofield of Battelle-Columbus contributed to the research.

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EXPERIMENTAL SCREENING OF CARBON-BASE MATERIALS
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INTRODUCTION

Since January, 1975, Battelle has been engaged in research on materials for improved impact members and heat shields in radioisotope heat sources to be used primarily for space power applications. The major goal of the research is to develop and demonstrate a bifunctional material that can perform advantageously as a combined impact member and heat shield, e.g., a bifunctional ablator. This report describes the screening studies done as an intermediate step to reach this goal and is essentially an interim summary of the program.

Previously reported work on the project included a comprehensive review and analysis of the state of developments in materials potentially suitable for impact member use^{(1)*}. Findings in that study led to the following conclusion:

"For a number of reasons, carbon-carbon composites as a class of materials currently appear to offer better prospects for impact member use than do metals, ceramics, or bulk graphites. However, carbon-carbon composites also present unique and complex technical problems in characterization, evaluation, and specification. Only limited knowledge is available with respect to preferred properties or structures for use in selecting or tailoring a composite specifically for impact member service."

Carbon-carbon composites also are prime candidates for improved heat shields, hence they have promise for meeting the goal of a bifunctional ablator material.

* References are given at end of report.

In the work being reported, an experimental approach was developed and used to evaluate various available carbon-carbon (C/C) composites for impact member use. Selected bulk graphites of interest for nose tips and heat shields of reentry vehicles were included in the experimental evaluations for comparison purposes.

In addition to the evaluations reported herein, special experiments were conducted to gain insight on the performance of the C/C composite impact member presently used in the most contemporary ERDA-developed heat source, the unit used in the multi-hundred watt (MHW) radioisotope thermoelectric generator (RTG). This RTG is serving in the near term as the power supply for selected DoD and NASA missions. These special experiments are the subject of a separate, concurrent report⁽²⁾, but salient data will be covered here for completeness.

The work was performed during the period from June, 1975, to July, 1976, and the data obtained are recorded in Battelle Laboratory Record Book No. 32008.

SUMMARY

Fourteen C/C composites and three reentry-grade bulk graphites were evaluated experimentally to determine their applicability for impact member use in radioisotope heat sources. The composites included the following generic types:

- (1) 2-D cloth lay-ups
- (2) 2-D and 3-D felts
- (3) 3-D weaves
- (4) 3-D pierced fabrics
- (5) 7-D weave
- (6) Coarse polar weave.

Also included was the 2-D randomly wound, resin-impregnated C/C material presently used as the impact member in the MHW RTG and commonly designated "GIS" (an acronym for graphite impact shell). While this material was evaluated primarily for the generic data to be obtained, it should be realized that considerable reference information exists (primarily empirical) for its impact performance.

The various materials were evaluated as energy absorbing materials using two fundamental types of tests. The first determined the energy associated with the formation of a projected area of new surface by a single fracture, e.g., work of fracture. In the other test, a relative measure of the energy absorbed in crushing, e.g., multiple fracturing, was determined by approximating the stress state encountered in impact member service where the member surrounds an essentially nonyielding fuel.

The ranking of materials from the results of both types of tests was essentially the same. Both precipitant and progressive failure modes were observed in both cases, with the preferable progressive mode exhibited by those materials with largest energy-absorption capacities.

None of the materials in these tests performed appreciably better than the GIS impact member material now used in the MHW heat source, HITCO Pyro Carb 814. Two cloth lay-up composites, HITCO's Pyro Carb 903 and Carborundum's Carbitex 700, were somewhat superior in performance, while the bulk graphites and felt-base composites ranked least effective as energy absorbers. All experimental data and other factors considered to date suggest that Pyro Carb 903 is the best prospect for a bifunctional heat shield and impact member. Its high density (1.80 g/cm^3) indicates potentially good ablation resistance to accompany its indicated good performance as an energy absorber.

BACKGROUND ON THE IMPACT MEMBER PROBLEM

The use of a radioisotope fueled heat source in space presents a potential hazard in that an aborted mission or orbital decay could result in earth reentry and ground impact. In order to assure survival during reentry and fuel containment on impact, a careful selection of heat source materials must be made early in mission planning so that appropriate safety margins can be incorporated into the design, and material properties in component configurations can be verified by test. This report focuses on evaluation of available materials in the C/C composite class that can assure containment in the event of earth impact of the next generation of isotopic heat sources.

Based on past and potential applications for isotopic heat sources, safety performance criteria were selected for impact velocity and temperature. For the former, it was decided that the impact material in component form should survive impacting an unyielding earth target at a velocity as high as 500 ft/sec. Although contemporary heat sources are designed to withstand impact at around 300 ft/sec, there are important improvements possible in heat source power density if a design can withstand higher terminal velocity. In order to have a variety of options, it might be desirable to have materials available which could withstand various terminal velocity regimes such as (a) <100 fps, (b) 100 to 300 fps, and (c) 300 to 500 fps.

In normal heat source operation the $^{238}\text{PuO}_2$ fuel surface is at temperatures ranging from 800 to 1300 C, and the impact member is 100 to 200 C lower in temperature. Further, the impact material must be sufficiently heat conductive for efficient heat transfer to externally located thermoelectric conversion elements or to a dynamic conversion system. However, during reentry it is desirable to have the impact member be poorly conductive in order to modulate the temperature of the primary fuel containment member. In practice to date, maximum power output from the RTG has demanded maximum temperature of the convertor and highly conductive impact member materials are used along with high melting point fuel container materials. Ingenious designs baffle the reentry heat, should reentry actually occur.

Figure 1 illustrates the general concepts of heat sources used in recent RTG's. Figure 1(a) shows the heat source used in the most recent modification of the SNAP-19 RTG. The cylindrical heat source capsule with hemispherical ends has an overall diameter of about 2.5 in., an overall length of about 5 in., and a weight of approximately 8 lb. This general design with only slight modifications is common to the Viking, Transit, and Pioneer RTG systems. The fuel in these systems is contained within a three-layer metallic shell with a 0.090-in.-thick layer of the tantalum alloy, T-111, as the strength member.

Figure 1(b) is representative of the fuel sphere concept most recently used in RTG's such as the multi-hundred watt (MHW) RTG being developed under ERDA sponsorship by General Electric. Twenty-four of these spheres are contained in a cylindrical graphite container which serves as an aeroshell for reentry protection. The $^{238}\text{PuO}_2$ fuel in this concept is positively contained

(a) SNAP 19 Capsule

(b) MHW Concept

(c) Multilayer Reference Heat-Source Configuration

FIGURE 1. HEAT SOURCE CONCEPTS

by a 0.020-in.-thick iridium clad and this in turn is surrounded by a 0.4-in.-thick 2-D C/C impact absorbing shell made by continuously winding a high-strength, high-modulus graphitic fiber yarn (Union Carbide Corporation's Thornel 50) on a spherical mandrel using a golf-ball winding machine to give a continually changing polar pattern. This yarn is then impregnated with a carbonaceous binder which is subsequently graphitized. A spherical threaded plug is then machined from the sphere, the mandrel removed, the fuel inserted, and finally the plug is replaced. This is the graphite impact shell (GIS) concept.

Figure 1(c) illustrates the approach currently being developed by ERDA where each $^{238}\text{PuO}_2$ fuel body will have its own containment, impact, and reentry protection as illustrated in the schematic cross section shown.

In general, on impacting an unyielding earth target, all or part of the kinetic energy of any heat source will convert to elastic strain energy in the impact member, and this energy will be distributed in a complex, rapidly changing stress field within the member. If the level of stresses is sufficiently low so that the material responds elastically, the heat source can simply recoil without being fractured or permanently deformed. From the standpoints of assured safety and ease of analytical prediction, this is the most desirable response. An analysis of possibilities for elastic response was the subject of an earlier project report⁽³⁾ which indicated that an impact member made from the best available candidate materials for providing this type of response (high-strength refractory ceramics) would limit impact velocities to less than 100 ft/sec.

If stresses exceed the strength of the impact member material, as has been observed in all service-type impact tests of heat sources to date, the kinetic energy can dissipate in either plastic flow or the formation of new surfaces by fracturing. For materials in which stresses upon impact exceed strengths, the best impact member material will be the one that absorbs the maximum energy per unit weight.

The current heat source used in space counts on protection in the event of impact by high strain rate mechanical crushing of the C/C composite impact shell. In this process extensive new surface is formed with a resultant large energy consumption. Since carbons and graphites exhibit brittle behavior in the impact member temperature regime, significant energy absorption by

plastic flow is not to be expected in C/C composites. It follows that emphasis in the investigation of these materials for impact member service must be placed on energy absorption in forming new surfaces by fracturing.

ENERGY ABSORPTION IN FRACTURING

As indicated above, the effectiveness of a C/C composite in impact member service will depend on energy consumed in the formation of new surfaces by fracturing. It is important, therefore, in the development, evaluation, and optimization of these materials to understand energy consumption by this process.

In general, if a local tensile stress in a brittle impact member exceeds the material's strength, the member can respond in either of two modes illustrated schematically in Figure 2.

Figure 2(a) represents a situation where the member fractures precipitantly in a few pieces, and because of the small area of new surface created, energy absorption is limited. This mode of failure is commonly observed in impact failures of low-porosity ceramics, including bulk graphites.

Figure 2(b) represents a different and highly preferable mode of failure in which the impact member crushes and energy is absorbed by multiple fracturing of a localized volume of material. The C/C composite impact member of the MHW heat source appears to behave in this manner in service-type impact tests. In these tests, however, the situation differed from that represented in Figure 2(b) in that only a portion of the available kinetic energy was absorbed in the impact member, resulting in severe deformation of the primary fuel containment shell. This is a higher risk situation that should be resolved for the next generation of heat sources.

The preferable crushing mode of failure shown in Figure 2(b) is probably associated with the existence of a crack-arrest mechanism in the material; i.e., once a crack is initiated, additional energy must be made available for its extension. Further, it is desirable that the crack extension should be localized within the central portions of the member and that very extensive breakup of the member should occur.

A number of local failure events can occur during the fracture of a generalized fiber composite. At some distance ahead of the crack, the fibers

FIGURE 2. BRITTLE MATERIAL RESPONSES TO IMPACT

are intact. In the high stress region near the tip, they are broken although not necessarily along the crack plane. Immediately behind the crack tip fibers pull out of the matrix, absorbing energy if the shear stress at the fiber-matrix interface is maintained while the fracture surfaces are separating.

In some bonded composites, the stresses near the crack tip could cause the fibers to debond from the matrix before they break. When total debonding occurs, the strain energy in the debonded length of fiber is lost to the material and is dissipated as heat. Fiber stress relaxation, a variation of the debonding model, indicates the elastic energy that is lost from a broken fiber when the interfacial bond is not destroyed. It is also possible for a fiber to be left intact as the crack propagates. This process, known as crack bridging, can also contribute to the toughness of the material. When brittle fibers are well bonded to a ductile metal matrix, the fibers tend to snap ahead of the crack tip leaving bridges of matrix material that neck down and fracture in a completely ductile manner. The fracture toughness in these circumstances is largely governed by the energy involved in the plastic deformation of the matrix to the point of failure. While all of these mechanisms may play different relative roles depending on the composite type, it is significant to note that no single mechanism can generally be assumed to account for the observed macro-fracture behavior of composites.

In terms of material properties, the ratio of the material's work of fracture to its fracture surface energy, γ_f/γ_i , must be greater than unity for crack arrest to occur. Work of fracture, γ_f , is the energy expended in creating a unity projected area of new surface by crack propagation, while fracture surface energy (or, simply, fracture energy), γ_i , can be interpreted as the energy per unit area required to initiate a crack. In a strict sense, γ_i , is the energy term in the Griffith⁽⁴⁾ criterion for crack instability*; i.e.,

$$\sigma_f = A \sqrt{\frac{2 \gamma_i E}{a}}, \quad (1)$$

* The original Griffith crack theory assumes that the only energy-absorbing process in fracture is the formation of new surfaces, and does not account for any plastic flow, fiber pullout, etc., at the crack tip.

where

σ_f = the applied stress at fracture

E = Young's modulus

a = depth of the critical flaw

A = a parameter dependent on flaw geometry and location.

A probable crack-arrest mechanism in C/C composites is energy dissipation from subsidiary crack branching around the tip of a primary crack as it moves through the material. If the energy consumed in forming these subsidiary cracks is the major factor causing γ_f to be larger than γ_i , the ratio γ_f/γ_i is an indicator of the extent of the subsidiary cracking as well as of the tendency of the material to crush. Further, if crushing is primarily the consequence of the formation of these branch cracks, a correlation would be expected between work of fracture and the total energy consumed when the material is crushed.

Since the Griffith criterion assumes the absence of a crack-arrest mechanism, an absolute value cannot be assigned to γ_i for a material that fractures progressively rather than precipitantly. However, the total energy consumed in fracturing a specimen, U_f , obtained from load-deformation data, can be measured and that portion of the energy consumed prior to a sharp drop in load, U_i , (if such occurs) taken as representative of the energy for crack initiation. In this approach, the ratio, U_f/U_i , from a standardized test is used for comparing materials with respect to crushing tendency, rather than the ratio of material properties, γ_f/γ_i .

Using this approach, if the specimen fails by a single primary crack, the material's work of fracture is simply $U_f/2A$, where A is the projected cross-sectional area of the specimen at the break. Work of fracture is a measure of energy-absorption capacity on an area basis, and thus relates only to two-dimensional material behavior, while in impact member service a volume of the material is crushed to powder. This situation imposed the need for additional data which BCL subsequently obtained. The new data reflect energy-absorption capacities of candidate materials on a volume basis, when each is crushed under stress-state conditions approximating those encountered in a heat source impact.

EXPERIMENTAL EVALUATIONS OF CARBON/CARBON COMPOSITES

On the basis of the above considerations, effort was directed to determining the following characteristics of available C/C composites to evaluate them for impact member service:

- (1) The total energy required to propagate a crack through a specimen under standardized conditions, U_f .
- (2) The material's work of fracture, $\gamma_f = U_f/2A$, where A is the projected fracture area.
- (3) The portion of the total energy stored at the time the primary crack begins to move through the specimen, U_i .
- (4) The total energy consumed in crushing (multiple fracturing) the material in a standardized test where the imposed stress state approximates an impact event.
- (5) The load at which crushing initiates in the multiple-fracture test.

In determining the above characteristics of various C/C composites, the tests reported here were conducted at room temperature and with slow loading rates ($\sim 7 \times 10^{-4}$ fps) relative to the rates of up to 500 ft/sec encountered in impact member service. This approach was taken for several reasons. First, the ability did not exist in the country for adequately instrumented testing to obtain the precise data required. Second, it was critical to obtain the maximum data possible on a comparative basis which could be done at the low strain rates. Finally, it was desirable to observe fracture patterns, which would have been impossible at the impact regime strain rates. Because strength of graphites and similar materials does not vary significantly with temperature up to the maximum temperature, i.e., 2500 F, of RTG operation, the expense of elevated-temperature testing was not assumed in early stages of the research.

Planned future investigations will examine in detail the effects of temperature and loading rates in the impact member service regimes on energy absorption in fracturing of selected C/C composites. However, it should be noted that strain rates as well as temperature have small effects on strength

and stress-strain characteristics of graphitic materials at temperatures below about 2300 C, suggesting that results of the room-temperature, low strain rate evaluations are valid for comparing the materials for impact member service.

Experimental Methods

Two test methods were used to determine the energy-absorption characteristics of the various carbon-base materials: (1) a work-of-fracture test for assessing U_f , U_i , and γ_f , and (2) a multiple-fracture test for assessing energy absorption under stress-state conditions approximating RTG impact where the fuel was considered essentially unyielding. In both techniques, a 20,000-lb, screw-loaded, standard Instron testing machine was used, and energy absorption in the specimen was determined from the area under the load-deformation curve recorded on the Instron chart.

Single edge-notched specimens were subjected to bending to determine the work of fracture.⁽⁵⁾ The specimen design, specimen dimensions, and loading configuration are shown in Figure 3. Specimens were loaded at a cross-head speed of 0.05 in./min. The energy absorbed prior to crack initiation, U_i , and to complete the fracture, U_f , was determined from respective areas under the load-deflection curve. The work of fracture, γ_f , was calculated from the equation, $\gamma_f = U_f/2B(W-a)$, where B, W, and a are as shown in Figure 3. Prior to gathering the data discussed in the body of this report, experiments were performed to prove the reproducibility of the technique, by developing values of U_f , U_i , and γ_i . These are described in Appendix A.

The multiple-fracture test was conducted by impressing a steel ball, 0.75 in. in diameter, into a 2 x 2 x 0.5-in. panel specimen, as shown schematically in Figure 4. A cross-head speed of 0.05 in./min also was used for this type of test. Load-deflection curves were recorded on an Instron chart, and the energy consumed in complete fracturing of each specimen was determined from the area under the curve. Also, the maximum load sustained by the specimen (P_{max}) was recorded as a measurement of the material's crushing strength.

Since only a spherical shape of the C/C composite material (HITCO 814) used in the MHW impact member was available, special experiments were required to obtain data comparable with that of the flat-panel specimens used for the other materials.

- S - Span = 1.5 inches
- B - Beam Width = 0.25 inch
- W - Beam Depth = 0.25 inch
- a - Notch Length = 0.125 inch
- c - Notch Width = 0.006 inch

FIGURE 3. WORK-OF-FRACTURE SPECIMEN AND LOADING CONFIGURATION

FIGURE 4. SCHEMATIC OF MULTIPLE-FRACTURE TEST

Materials Evaluated

Graphitic fibers in the form of multistrand yarns are used in several basic configurations to make C/C composites by

- (1) Non-intertwined windings,
- (2) Cloth or felt lay-ups, or
- (3) Intertwined weaves or felts.

The latter provides multidirectionality of the yarn and a less anisotropic structure. A wide variety and combination of each of these general configurations are possible, and a variety of fibers and yarns is available from which to make composites of various properties. In addition, the properties of a composite can be modified by the type and amount of matrix carbon in the structure. The matrix carbon is introduced either as a sizing applied to the yarn prior to winding or weaving which is subsequently charred, or by impregnation of the shaped yarn article with a carbonaceous liquid or vapor.

The Pyro Carb 814 composite used in the MHW heat source impact member represents a typical laminar, wound configuration. It was of particular interest in the present investigation to determine whether felted-yarn, cloth lay-ups, or woven multidirectional yarn structures offer improved energy-absorption characteristics than the MHW impact member configuration. Each of these other configurations is being considered in Department of Defense programs for reentry nose cone and heat-shield applications, and their availability results from this interest rather than from any established commercial use. With cooperation from DoD agencies, samples of C/C composites having the other yarn configurations were obtained for energy-absorption testing. Table 1 lists the C/C composites tested; the supplier, bulk density, yarn configuration, and fiber precursor are also shown. In addition, three high-density, fine-grained bulk graphites of interest for reentry nose cones and heat shields were selected and tested.

Results of Tests on Carbon/Carbon Composites

Table 2 summarizes the data obtained from both the work-of-fracture and multiple-fracture tests on the selected materials. For the anisotropic materials (e.g, cloth lay-ups), the data in Table 2 are for the direction giving highest energy-absorption values, which was in all cases with the load applied normal to the principal fiber orientation.

TABLE 1. CARBON-BASE MATERIALS EVALUATED FOR ENERGY-ABSORPTION CHARACTERISTICS

Material	Supplier	Grade	Density, g/cm ³	Yarn Configuration (1)	Fiber Precursor
ATJ	Union Carbide	ATJ-S	1.83	Bulk graphite	
POCO	POCO Graphite	AXF-5Q	1.83	"	
Graphnol	ORNL	N3	1.85	"	
Pyco Bond	Super Temp	407-80	1.82	3-D felt	Rayon
"	"	807-82	1.60	2-D felt	Rayon
"	"	418-80	1.75	2-D knit	PAN
Pyro Carb	HITCO	903	1.80	2-D cloth lay-up	PAN
"	"	508	1.35	2-D cloth lay-up	Rayon
3-D weave	FMI	PN 491B	1.90	3-D weave (2,2,3)	Rayon
"	"	PN 557B	1.90	3-D weave (2,2,4)	Rayon
Pierced fabric	AVCO	MOD 3A	1.60	3-D pierced fabric	Rayon
"	"	MOD 3B	1.20	3-D pierced fabric	Rayon
3-D weave	GE	3-D weave	1.85	3-D weave (2,2,3)	Rayon
7-D weave	GE	7-D weave	1.90	7-D weave	Rayon
3-D cylinder	GE	Polar weave	1.85	Coarse polar weave (12,4,4)	Rayon
Carbitex	Carborundum	700	1.46	2-D cloth lay-up	Rayon
Pyro Carb	HITCO	814	1.20	2-D randomly wound sphere (2)	Rayon

(1) 2-D, 3-D, and 7-D refer to the number of directions in which the yarn is oriented in the structure.

(2) MHW heat source impact member material.

TABLE 2. SUMMARY OF ENERGY-ABSORPTION CAPABILITY BY CARBON-CARBON COMPOSITES

Material	Fracture Mode	Work-of Fracture Test					Multiple-Fracture Test			Note
		Maximum Load, lb	Energy Absorbed, in.-lb		Work of Fracture, γ_f , in.-lb/in. ²	Maximum Load, lb	Total Energy Absorbed, in.-lb			
			Total, U_f	Initiation, U_i						
					U_f/U_i					
<u>Bulk Graphites</u>										
Union Carbide ATJ-S	Precipitant	7.5	0.031	0.015	2.07	0.50	Precipitant	2000	<100	
PCCO AXF-5Q	Precipitant	12.0	0.033	0.027	1.22	0.53	Precipitant	3200	<100	
ORNL Graphnol N-3	Precipitant	9.0	0.045	0.040	1.13	0.72	Precipitant	5800	370	
<u>Cloth Lay-Ups</u>										
Super Temp Pyco Bond 418-80	Precipitant	18.5	0.91	0.22	4.24	14.60	--	--	--	2-D knit
HITCO Pyro Carb 508	Progressive	13.6	1.15	0.19	6.05	18.40	Progressive	4275	1150	
HITCO Pyro Carb 903	Progressive	14.0	0.80	0.13	6.15	12.80	Progressive	6250	1725	
Carborundum Carbitex 700	Progressive	25.0	2.06	0.35	5.89	33.00	Progressive	9500	2000	
<u>Felts</u>										
Super Temp Pyco Bond 407-80	Precipitant	13.1	0.13	0.05	2.37	2.05	Precipitant	5300	325	3-D
Super Temp Pyco Bond 807-82	Precipitant	11.5	0.08	0.03	2.56	2.31	Precipitant	5100	142	2-D
<u>Multidirectional Weaves</u>										
FMI 3-D Weave PN 491B	Precipitant	44.5	0.75	0.28	2.68	15.00	--	--	--	2-2-3 weave
FMI 3-D Weave PN 557B	Precipitant	27.5	0.53	0.26	2.04	10.50	--	--	--	2-2-4 weave
AVCO Pierced Fabric MOD 3A	Mixed ⁽¹⁾	23.0	0.63	0.23	2.74	10.08	Precipitant	6000	450	
AVCO Pierced Fabric MOD 3B	Progressive	9.0	1.23	0.26	4.73	19.68	Progressive	3200	1180	
GE 3-D Weave	Mixed ⁽¹⁾	62.5	0.98	0.52	1.88	15.68	Mixed ⁽¹⁾	6850	665	2-2-3 weave
GE Polar Weave	Precipitant	33.2	0.55	0.45	1.22	8.80	--	--	--	12-4-4 weave
GE 7-D Weave	Progressive	32.5	1.05	0.25	4.20	16.80	Precipitant	9500	1040	
<u>MHW Impact Member - Wound Sphere</u>										
HITCO Pyro Carb 814	Progressive	11.0	~2.00	~0.05	~40.0	30.00	Progressive	3350	1520 ⁽²⁾	

(1) Biased slightly toward progressive mode.

(2) See Appendix B.

As mentioned, two kinds of fracture modes were noted in each test for the materials, precipitant and progressive. Typical load-deflection curves for each mode are shown in Figures 5 and 6. With the precipitant mode, the load dropped rapidly to zero after the inception of crack instability at maximum load. For specimens which exhibited the progressive mode of failure, the shape of the curve makes it obvious that cracks propagated in several stages of alternate crack arrest and extension, and underwent considerable deformation prior to the load gradually dropping to zero.

Figure 7 suggests that the energy absorbed in the multiple-fracture tests generally is proportional to that absorbed in the work-of-fracture test. This, in turn, suggests that energy dissipated by subsidiary crack branching in the vicinity of the primary crack tip constitutes the principal crack-arrest mechanism in these carbon-base materials as postulated earlier.

The fact that Pyro Carb 903 shows the largest deviation from the proportionality indicates that the fracture modes and/or energy-absorption mechanisms in the two tests are different for this material. Although fracture modes were progressive in both the tests, a visual inspection of the fracture surfaces indicated that the energy absorption in the work-of-fracture test was predominantly by delamination of layers stacked perpendicular to the load axis, while that in the multiple-fracture test was by both delamination of stacked layers and crushing of materials in individual layers. An additional energy absorption by crushing, therefore, appears to account for a higher ratio of energy absorption in the multiple-fracture test and that in the work-of-fracture test.

In addition to the relationship above, it can be seen in Table 2 that the mode of failure, i.e., precipitant or progressive, was nearly independent of the type of test for any given material. These similarities indicate that either test could be used to screen materials for energy absorption and tendency to crush in impact member service. Also, it will be noted that the progressive mode of failure, indicating crushing capability, tended to accompany high values of energy absorption as well as high U_f/U_i ratios.

A major finding in these experimental evaluations is that none of the various carbon-base materials offer much advantage over the presently used HITCO 814 spherical wound C/C composite impact member material in energy-absorption capacity. The two cloth lay-up composites, HITCO's Pyro Carb 903 and Carborundum's Carbitex 700, performed about equivalent to the HITCO 814.

FIGURE 5. TYPICAL LOAD-DEFLECTION CURVES FROM THE WORK-OF-FRACTURE TEST

FIGURE 6. TYPICAL LOAD-DEFLECTION CURVES FROM THE MULTIPLE-FRACTURE TEST

FIGURE 7. RELATION OF ENERGIES ABSORBED IN WORK-OF-FRACTURE AND MULTIPLE-FRACTURE TESTS

Three materials--a cloth lay-up (HITCO's Pyro Carb 508) and two with multi-directional fiber weaves (AVCO's MOD 3B and GE's 7-D)--performed considerably better than any of the remaining materials. Clearly, the bulk graphites and felt-base C/C composites were inferior in performance in these tests.

Of the materials that performed well in these evaluations, two have relatively high bulk densities--HITCO's Pyro Carb 903 (1.80 g/cm^3) and GE's 7-D (1.90 g/cm^3)--which is the usual prerequisite for good ablation resistance and, hence, may be candidates for a bifunctional ablator.

EFFECT OF STRAIN RATE ON ENERGY ABSORPTION

It is emphasized that the above data are from tests conducted at room temperature and at low strain rates when compared to actual RTG service conditions. Based upon available literature indications that strain rate and temperature variations will not affect the test results in the regimes of interest, this approach was warranted. In an initial attempt to verify the insensitivity to strain rate variation of energy absorption, work-of-fracture tests were conducted on one of the C/C composites (Pyro Carb 508) and one of the bulk graphites (ATJ-S) at strain rates over the range available with the Instron test machine (0.005 to 0.5 in./min). The results obtained by this two-decade variation of strain rate are shown in Figures 8 and 9 and indicate that there is essentially no effect of strain rate within that range on either the energy-absorption capacity or the fracture characteristics of these materials. That is, the fracture mode for the ATJ-S and Pyro Carb 508 remained precipitant and progressive, respectively, as shown by the shape of the curves. As pointed out earlier, the 0.5 in./min rate ($\sim 7 \times 10^{-3}$ fps) is still approximately 5 orders of magnitude lower than the maximum possible impact velocity of 500 fps, so that prudence suggests that verification of the lack of strain rate effect be extended to that range. BCL has now essentially completed the assembly of a sophisticated device to obtain load-deflection curves for materials impacted in the 300 to 500 fps range and will soon investigate this factor.

Regarding the effect of temperature, the literature is more substantive in indicating that mechanical properties of graphites and C/C composites vary only in a marginal manner as a function of temperature up to values much in excess of the approximate 2500 F range expected for RTG operation. However, this factor will be verified in the near future for work-of-fracture values.

FIGURE 8. LOAD-DEFLECTION CURVES SHOWING NEGLIGIBLE STRAIN RATE EFFECTS IN PYRO CARB 508

FIGURE 9. LOAD-DEFLECTION CURVES SHOWING NEGLIGIBLE STRAIN RATE EFFECTS IN ATJ-S

CONCLUSIONS

These studies have generated a basic understanding of energy-absorption capacity and failure mode of C/C composites and graphites which will be expanded upon to select an improved impact member for RTG's. In general, the most promising class of materials from an impact absorption viewpoint is the C/C composites, especially the two-dimensional materials oriented with the principal weave perpendicular to the direction of impact loading. Within the range of strain rates tested (0.005 to 0.5 in. per min) there was no effect of this variable. The most promising material currently available commercially for a bifunctional ablator seems to be Pyro Carb 903, based on its combined fairly high density and high energy absorption in crushing.

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APPENDIX A

REPRODUCIBILITY OF THE WORK-OF-FRACTURE TEST

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REPRODUCIBILITY OF THE WORK-OF-FRACTURE TEST

Because the work-of-fracture test measures the energy absorbed in propagating a single primary crack originating at notch tip, there was concern that the notch-tip site with respect to microstructural features of the C/C composite might affect test results. To check this possibility, randomly notched specimens of Carbitex 700 were evaluated. Load-deflection curves for seven specimens are shown in Figure A-1. Notches in the four specimens of Figure A-1(a) were cut perpendicular to those in the three specimens of Figure A-1(b).

As can be seen from Figure A-1, the results of the tests were quite reproducible, indicating little or no effect of notch tip. Data from Figure A-1 are tabulated below.

	<u>Figure A-1(a)</u>	<u>Figure A-1(b)</u>
Maximum load, lb	25.0 $\pm$ 2.0	30.8 $\pm$ 0.8
Total energy absorbed, in.-lb	2.06 $\pm$ 0.27	1.45 $\pm$ 0.05
Work of fracture (γ_f), in.-lb/in. ²	33.0 $\pm$ 4.0	23.0 $\pm$ 0.8

FIGURE A-1. CARBITEX LOAD-DEFLECTION CURVES SHOWING REPRODUCIBILITY OF WORK-OF-FRACTURE TEST DATA

APPENDIX B

COMPARATIVE MULTIPLE-FRACTURE DATA ON MHW IMPACT MEMBER MATERIAL

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COMPARATIVE MULTIPLE-FRACTURE DATA ON MHW IMPACT MEMBER MATERIAL

A multiple-fracture test on a spherical section cut from HITCO 814 impact shell used in the MHW heat source was conducted utilizing uniaxial loading with a 0.75-in. steel ball as in the standard multiple-fracture test. The specimen was supported on a spherical mandrel as shown in Figure B-1. The total energy absorbed by the specimen as determined from the area under the load-deflection curve was 950 in.-lb.

Because of differences in imposed stress state, the energy absorbed by this spherical segment is not directly comparable to energies absorbed in the standard multiple-fracture tests on flat panel specimens of other materials reported in Table 2 of the body of this report. To account for the sphericity effect, two additional multiple-fracture tests were conducted using specimens of Carbitex 700. In one test, the specimen geometry and loading configuration were identical to the above test of HITCO 814. The other was a standard multiple-fracture test of a flat panel specimen. Load-deflection curves gave absorbed energies of 1250 and 2000 in.-lb, respectively. In post-test examinations, all three specimens exhibited similar crushing modes of failure.

Because of the similarity in fracture mode and other responses, a flat panel specimen of HITCO 814, if available, should absorb more energy than that measured for the spherical-segment specimen (950 in.-lb), and the amount would be expected to be approximately proportional to that found for Carbitex 700 specimens (2000:1250). On this basis, the comparable energy absorption of a flat panel of HITCO 814 was calculated to be $950 \times 2000/1250 \approx 1520$ in.-lb. This is the value recorded in Table 2.

FIGURE B-1. MULTIPLE-FRACTURE TEST OF MHW IMPACT SHELL SEGMENT

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